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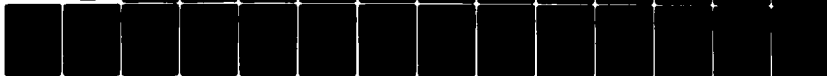
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MICROSTRUCTURAL DEVELOPMENT AND EVOLUTION  
DURING LIQUID PHASE SINTERING

FINAL REPORT

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T. H. COURTNEY AND J. K. LEE

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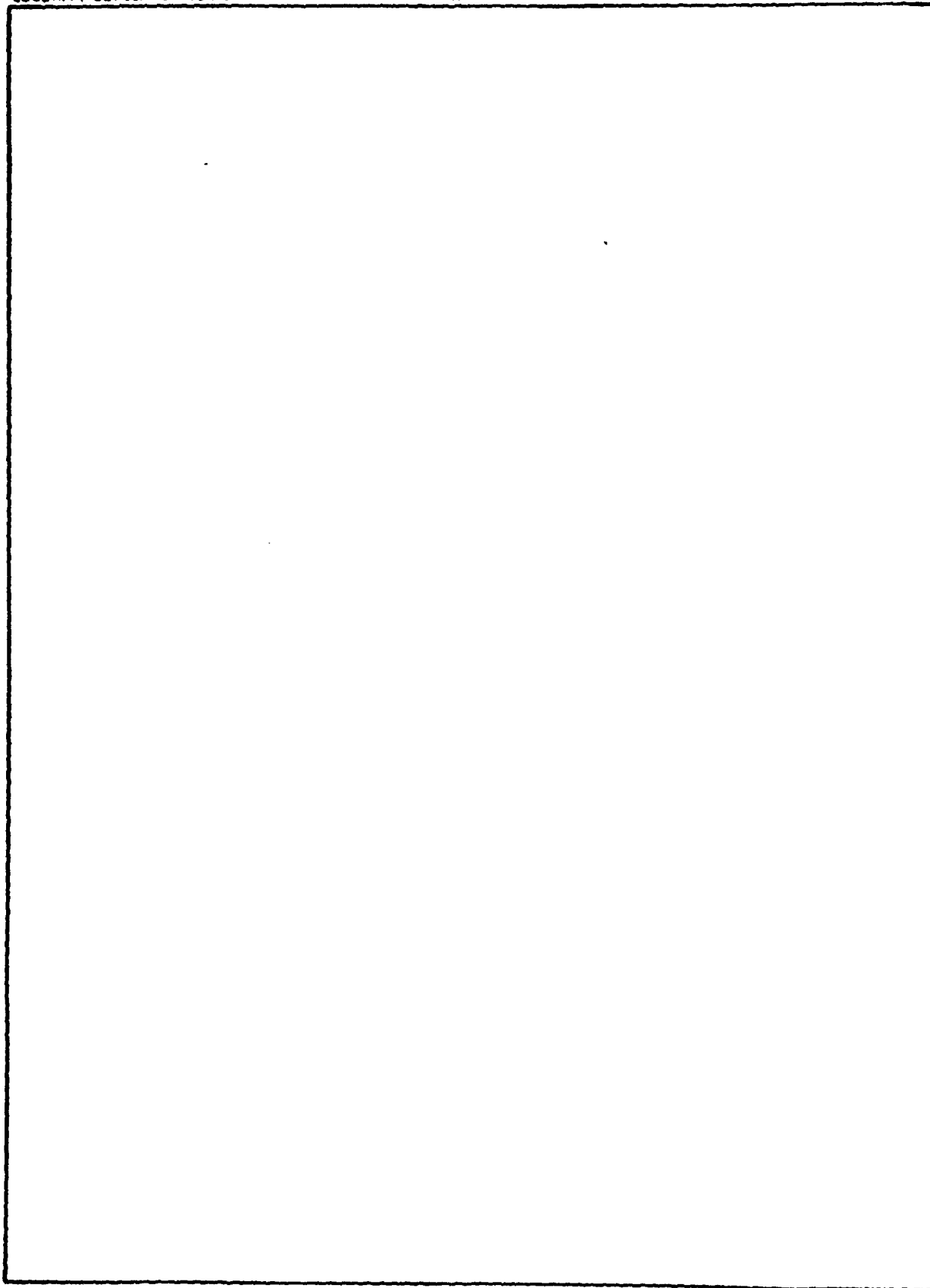
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Microstructural development and evolution during the liquid phase sintering process has been studied in terms of thermodynamic, kinetic and structural features. Solid phase continuity primarily depends on solid volume fraction. Microstructural scale is adversely affected by continuity since, under this condition, coarsening is accelerated by particle coalescence. It is found that continuity can be expressed as a single function of particle volume fraction and microstructural scale can be adequately related to system parameters as well as particle volume fraction.		

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Microstructural Development and Evolution  
During Liquid Phase Sintering

Final Report to Army Research Office  
Grants Nos. DAAG29-77-G-0235  
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Principal Investigators: T. H. Courtney and J. K. Lee

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A

I. Introduction

The objective of the research supported by these grants is to clearly define the structural and chemical factors influencing the scale and contiguity of the solid phase present during liquid phase sintering (LPS). Because of complexities attendant with initial densification in such materials, we have studied scale and contiguity in alloys sintered for times in excess of those required to achieve full density. By so doing, we feel we have been able to more clearly delineate the important physiochemical and geometrical parameters affecting microstructural development and evolution.

Optimum mechanical properties of LPS alloys are generally associated with 1) a high volume fraction ( $V_p$ ) of the phase remaining solid during the sintering process, 2) a fine structural scale of this phase, and 3) low contiguity of the solid phase. The first two of these features are associated with increased strength and hardness of the alloy whereas the latter is usually associated with improved fracture toughness. In the presentation following, we discuss the interrelationships between these three structural features. Much of the work described has been published or submitted and will be presented in a summary type manner without detailed discussion. The work that has not yet been submitted for publication will be presented in greater detail.

In addition to increases in fracture resistance associated with low

solid phase contiguity in LPS alloys, low contiguity also has potential use in subsequent materials processing. We have attempted to capitalize on this by mechanical production of fibrouslike composites directly from LPS alloys of low solid phase contiguity. We have had some success in this regard in the sense that we have produced strong ultrafine fibrouslike composites of iron in copper by extensive mechanical deformation of LPS alloys. We do not know yet just what structural features ( $V_p$ , contiguity, etc.) allow for such deformation, nor do we know in detail the relative contribution of the various hardening mechanisms (cold work, fiber, size effects, etc.) to the strength of the materials so produced. Because of this, no papers have yet been published in this area but we do have a reasonable amount of data on hand and these, along with our present interpretation of their meaning, will be presented in reasonable detail in this report. The report itself is divided into two categories: 1) "Microstructural Development and Evolution" (the area in which we have spent most of our time and effort), and 2) "Mechanical Processing of Liquid Phase Sintered Alloys."

## II. Research Accomplishments

### A. Microstructural Development and Evolution

As a result of extensive experimental studies on the Fe-Cu (1,2) the W-(Fe-Ni-W) (2) and lesser work on the MgO-(MgCaSiO<sub>4</sub>) systems (2), we believe we have demonstrated that particle contacts occur sufficiently frequently during LPS so that physical and chemical features of the system play a secondary role in determining particle contiguity. Instead  $V_p$  is the main factor in determining solid phase contiguity. This is shown in Fig. 1 where—  
in the number of particle-particle contacts in a two-dimensional plane of polish,  $C_p$ ,\* a measure of particle contiguity, is plotted as a function of  $V_p$ .

\*In determining particle-particle contacts, particle coalescence is taken into account by arbitrarily assuming two particles have fused into one when the ratio of the two-dimensional neck between the particles to the two-dimensional radius of the smaller of the contacting particles exceeds two-thirds.

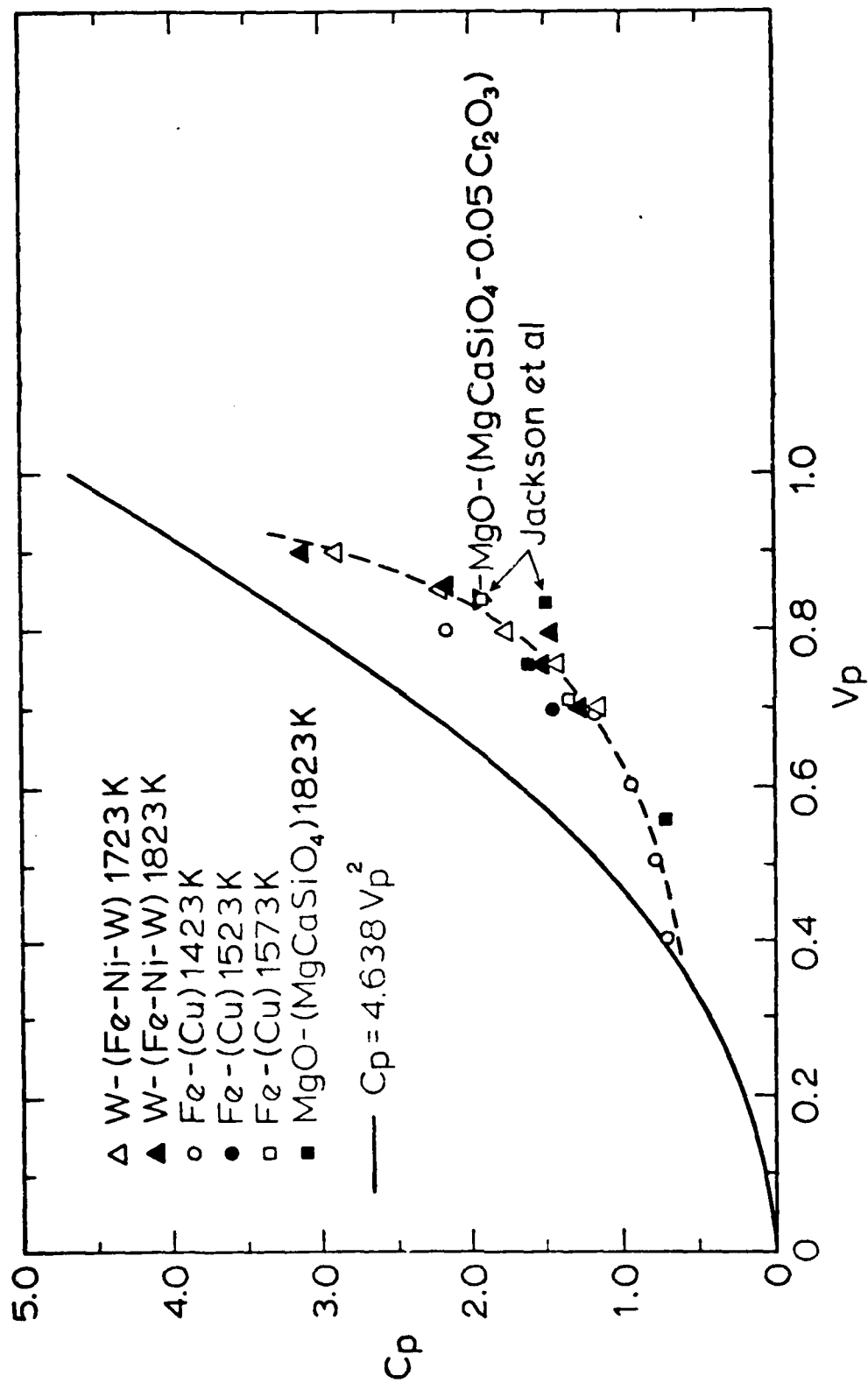


Figure 1



for the several LPS systems studied. The solid line shown in Fig. 1 is obtained through a numerical simulation applied to a nonwetting system. That is, for the alloys studied, the reduction in  $C_p$  obtained in comparison to the solid line represents the effect of wetting on  $C_p$ . Values of  $C_p$  shown are "steady state" values. That is the values shown are ones obtained over a broad range of sintering times. Moreover, the  $C_p$  values are also independent of the sintering temperatures employed in this study. That a steady state microstructure is obtained is not surprising for a microstructure in which only the scale and not the contiguity changes with increased sintering time is not only physically appealing but is also plausible based on theoretical grounds. On the other hand, that  $C_p$  is independent of sintering temperature is somewhat unexpected and what is especially intriguing is that contiguity is system independent, depending only on  $V_p$ . In essence, these data signify to us that frequency of interparticle contact is sufficiently high so that the structural feature,  $V_p$ , determines contiguity to a much greater extent than do the differing physiochemical characteristics of the studied systems.

What causes particles to make contact during liquid phase sintering? Neglecting thermal convection (presumably absent in our studies) we can think of two reasons: particle Brownian motion and gravity induced contacts (3). Calculation and experimental observations (1) lead us to believe that, except for very fine particle dispersions (particle size at least less than 1  $\mu\text{m}$ ), gravity differences between the liquid and solid account for most particle contacts. Since a three-dimensional skeletal type structure of the solid obtains when  $C_p \geq 0.75$  (1), gravity induced preferential migration of one phase will not lead to appreciable macrosegregation in the systems studied (see Fig. 1). However, when  $V_p$  is sufficiently low, perceptible settling of LPS compacts occurs. From a technological processing point of view, therefore,

establishment of solid state contiguity is beneficial since it prevents macro-segregation from occurring on the same time scale as sintering.

There still remains a dichotomy in LPS alloys. That is, in common with almost all LPS systems, the alloys we studied are "wetting" systems. Yet apparently subsequent to densification solid particles move about within the liquid and collide. A collision which leads to a particle contact is a priori evidence of a local reduction in the surface energy, i.e. the solid surfaces making contact do not fulfill the wetting condition. Since, on a macroscopic scale, the system is wetting, we have assumed that the boundary between two solids that stick subsequent to a collision is a low angle, low energy grain boundary. In order to obtain an estimate as to the frequency that a particle collision will lead to a low angle boundary being formed, we performed a simple analysis based on probabilistic considerations (4). In effect, we assumed the surface of a spherical particle to be comprised a large number of specific crystallographic planes. The exact number of planes on the surface is related to the width of the energy cusp at a low angle boundary. If the width of the cusp is small, the number of planes on the surface is assumed to increase. The probability of a given collision leading to a low angle boundary is related to the square of the fractional surface area occupied by  $\{hkl\}$  planes, multiplicity being explicitly considered. For example, the probability of  $\{123\}$  planes making contact is proportional to the square of the surface area of the sphere belonging to all planes of the  $\{123\}$  type. The total coalescence probability,  $P_c$ , is then obtained by summing up over all planes on the surface. The above procedure describes the basis for calculation of  $P_c$  by the "finite orientation counting" method (Fig. 2). Monte Carlo simulation was also used to model this process and the results of this procedure, along with a simple derived analytical relationship, are also shown in Fig. 2. At

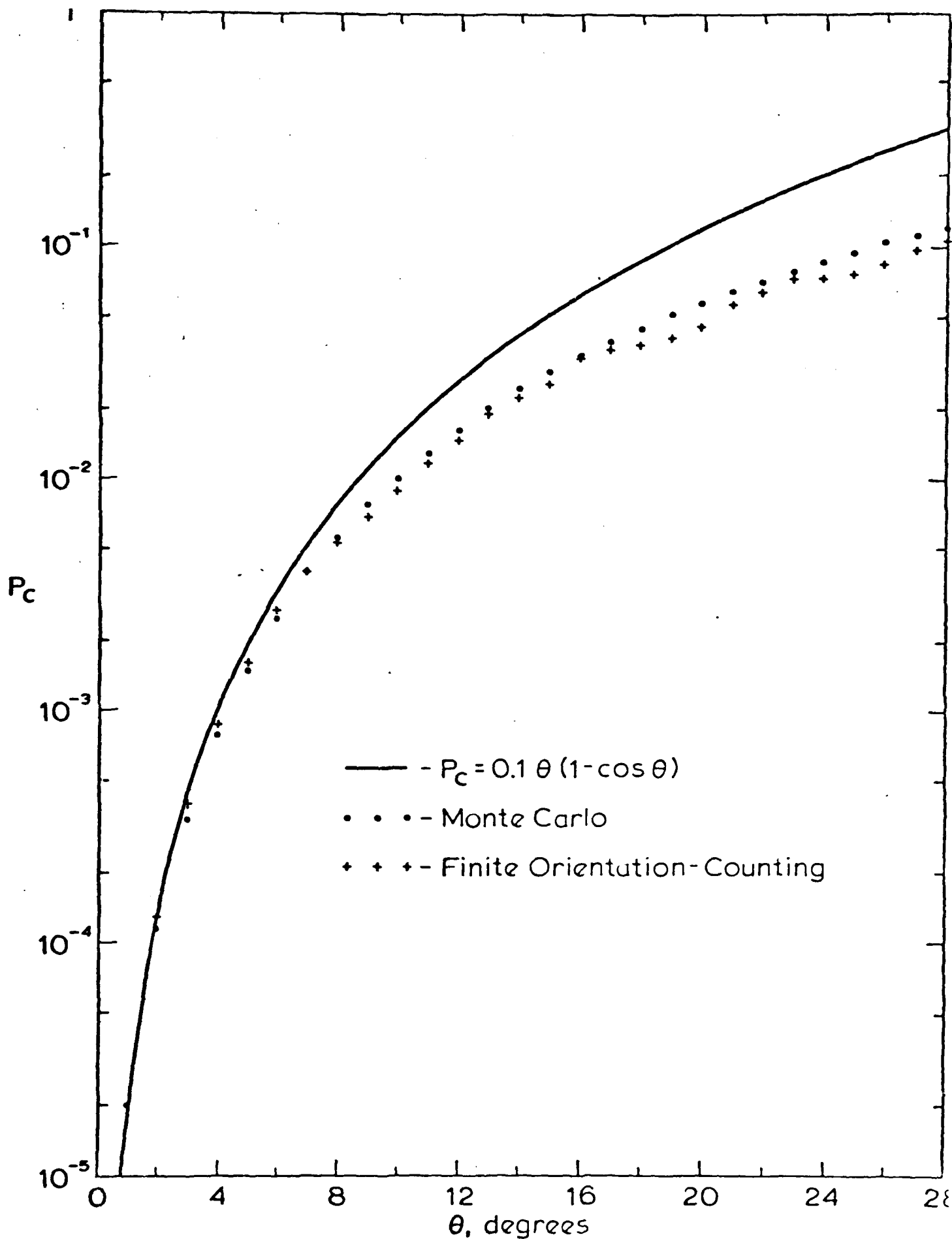


Figure 2

low values of the parameter  $\theta$  ( $= \frac{2\gamma_{SL}}{\gamma_{SS}} \phi$  where  $\gamma_{SL}$  = solid-liquid surface energy,  $\gamma_{SS}$  = high angle grain boundary energy and  $\phi$  = width of energy cusp), applicable to most LPS systems, the results of all three methods of calculation are essentially equivalent. Although the results of such an approach are only as good as the approximations utilized, we believe they are useful in defining how structural ( $\phi$ ) and chemical ( $\gamma_{SL}/\gamma_{SS}$ ) features affect  $P_c$ . Moreover, we believe the numbers calculated serve as an order of magnitude estimate on  $P_c$  and, in this regard, are important in that  $P_c$  is low ( $\leq 10^{-3}$ ) for a "typical" LPS system. That a large number of sticking contacts is made even with small values of  $P_c$  is further indirect evidence that a large number of contacts is made during LPS.

This theoretical result served as the basis for an experimental study, the results of which, to date, are unfortunately ambiguous. Specifically, we have taken our liquid phase sintered alloys and are attempting to find the orientation relationships between particles that have coalesced during the sintering process. This is being pursued by electron channeling analysis of adjacent grains. There is, as far as we are aware, one micrograph in the literature (5) which supports our main contention and utilizes the channeling technique and therefore suggests we are on the right track. However, as noted, our results are ambiguous and, in the last part of this section, we present recent results which suggest the cause of this ambiguity.

Microstructural scale depends on two processes. One, Ostwald ripening, refers to the process wherein mass is transferred, through the liquid, from a smaller than average size particle to one with a size greater than average. During the process, the solid-liquid interface area continually decreases and when the small particle is dissolved, the system has "lost" one particle. The other process is coalescence-fusion. After a contacting collision is

made, mass is transported, again through the liquid, from the circumferences of the contacting particle to the neck at the contact point. During this process, surface area is also continually reduced and, when the neck is "filled in," a particle is also removed from the system. Both processes operate concurrently and hence both contribute to microstructural coarsening. If  $S_v$  is the particle-liquid interfacial area per unit volume (note that for a given  $V_p$ ,  $1/S_v$  is a measure of particle "size,"  $r$ ), the time variation of  $S_v$  can be expressed as (1,2)

$$\frac{1}{S_v^3} - \frac{1}{S_{v0}^3} = \frac{1}{27V_p^3} \frac{t}{\tau_c}, \quad (1)$$

where  $S_{v0}$  is the original particle-liquid interfacial area per unit volume. The time constant,  $\tau_c$ , in general incorporates both coarsening processes. If Ostwald ripening alone is dominant then  $\tau_c = \tau_{or}$ , the Ostwald ripening time constant. On the other hand, if both Ostwald ripening and coalescence-fusion contribute substantially to coarsening  $\tau_c = \tau_{or}\tau_f/\tau_{or} + \tau_f$  where  $\tau_f$  is the time constant for the coalescence-fusion process. A theoretical basis for the  $V_p$  dependence of  $\tau_{or}$  (6) and  $\tau_f$  (1,7) has been developed. Figure 3 shows the  $V_p$  dependence of the experimentally measured values of  $\tau_c$  for the Fe-Cu system along with the expected variation of  $\tau_{or}$  and  $\tau_{or}\tau_f/\tau_{or} + \tau_f$  with  $V_p$ . In constructing these curves, one datum point, at  $V_p = 0.40$ , has been force fitted to the  $\tau_{or}$  curve. Even though force fitting of one datum point is not appealing, the data clearly illustrate that the  $V_p$  dependence of  $\tau_c$  is much greater than the expected dependence of  $\tau_{or}$  or the time constant for parallel processes on  $V_p$ . Hence we conclude that a transition in the coarsening mechanism occurs at  $V_p \approx 0.40$ . For values of  $V_p$  less than 0.40, Ostwald ripening is the main contributor to coarsening. For values of  $V_p$  in excess of this, both Ostwald ripening and coalescence-fusion contribute to coarsening.

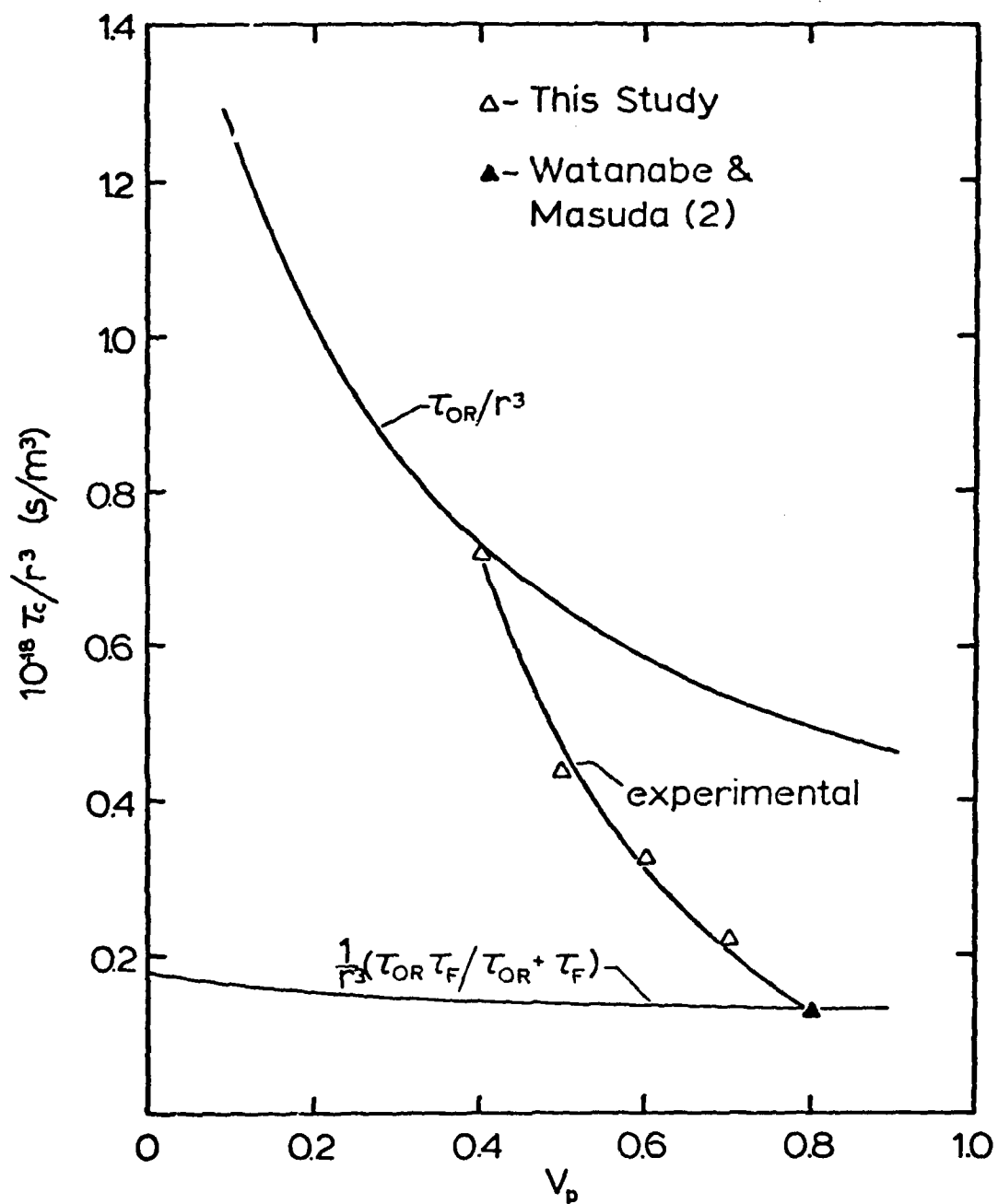


Figure 3

This interpretation is reinforced, moreover, by the fact that the transition occurs in the vicinity of  $V_p \cong 0.40$  which corresponds approximately to a transition from a skeletal, contiguous solid phase to an isolated one (1). This situation is not unique to the Fe-Cu system as is shown in Fig. 4 which includes measured values of  $\tau_c$  for other systems (2). The precipitous decrease in  $\tau_c$  with small increases in  $V_p$  in the region where a skeletal structure is forming rapidly is theoretically consistent only with a transition in coarsening mechanism. Hence we conclude that increases in  $V_p$  in LPS alloys not only lead to increases in contiguity but coarser microstructures as well. From this we can state that the desirous aim of producing high volume fraction solid phase composites with low contiguity and fine scale is basically fighting against nature. Success in so doing can come about only as a result of creative thinking and/or serendipity.

Ostwald ripening and coalescence fusion are driven by the same thermodynamic driving force and controlled by the same kinetic processes. Only the local and microscopic geometry affect the rates of these processes differently. The kinetic factors controlling coarsening in each are the solubility of the solid phase in the liquid and the temperature; the latter does so through the inverse temperature dependence associated with all kinetic processes and through the temperature variations of the liquid diffusivity. The wide system-to-system variation of  $\tau_c$  illustrated in Fig. 4 can be partially removed by a normalization process which takes into account these factors (2). This is done in Fig. 5. The remaining scatter in the data is due to system-to-system variation in the solid-liquid surface energy and by the approximation of the effect of temperature on the diffusivity by a simple superheat term  $(\Delta T/T_m)$ . (Implicit in the normalization procedure is the reasonable, although not correct, assumption that liquid diffusivities are the same at the melting tempera-

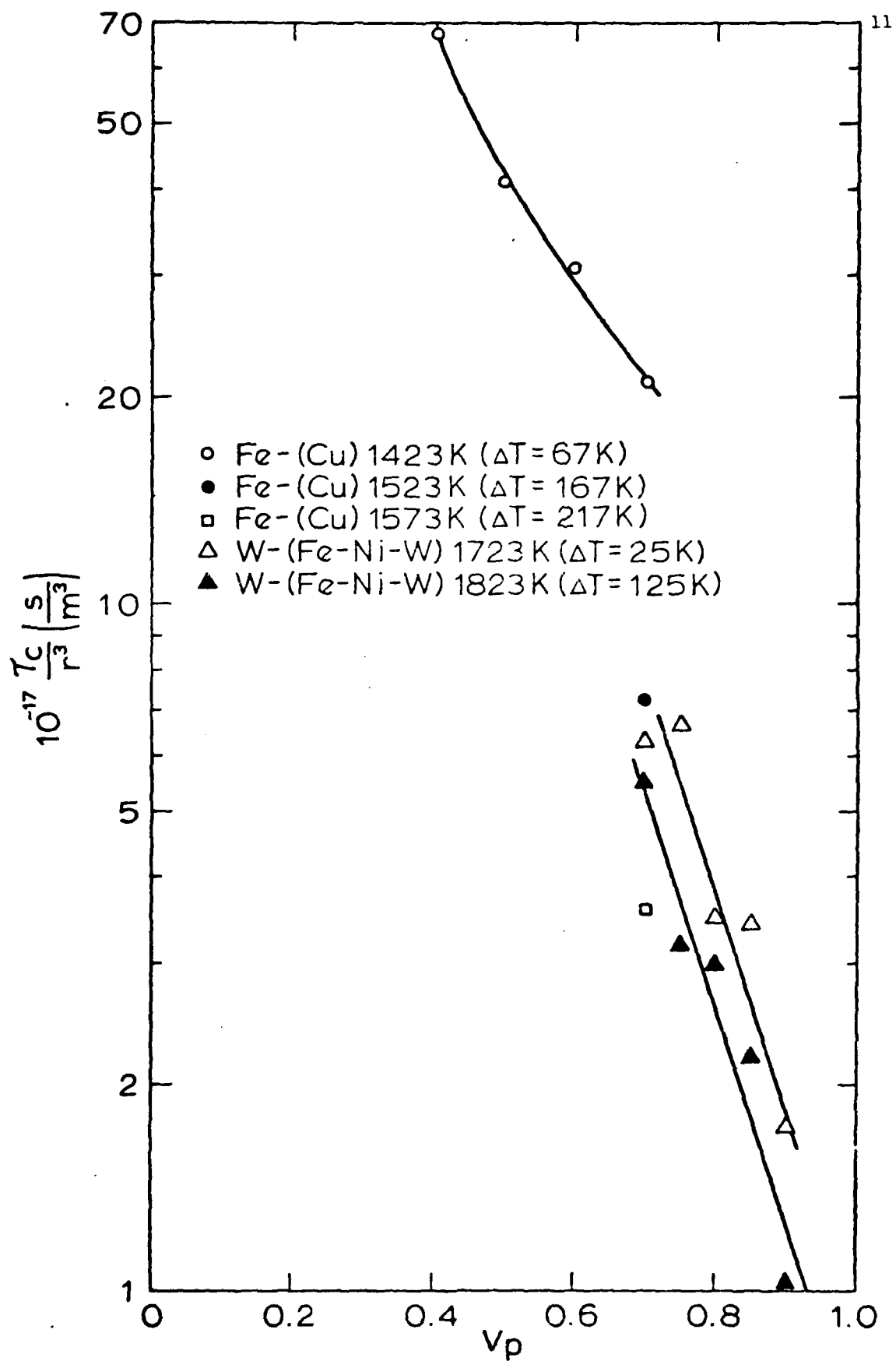


Figure 4



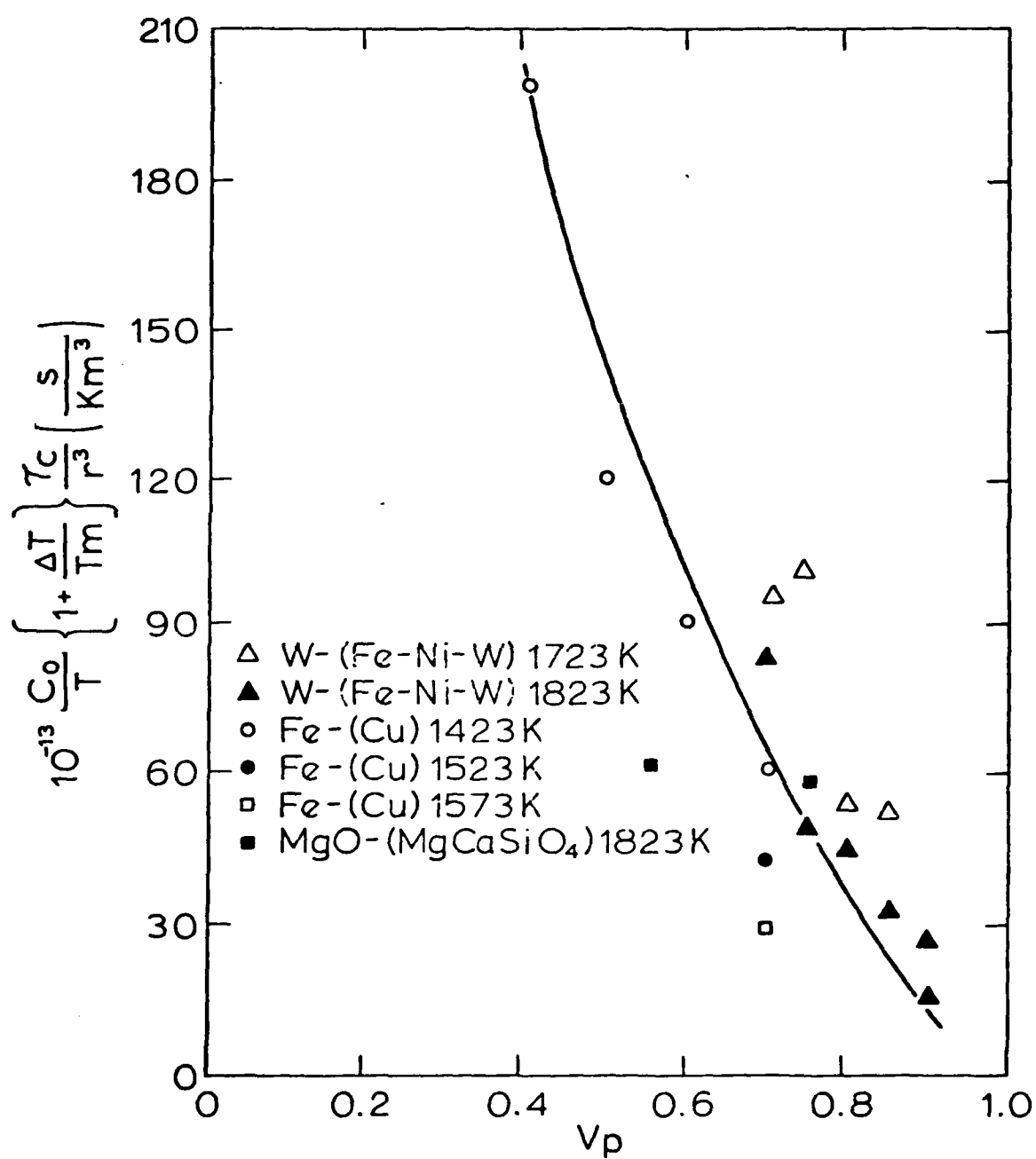


Figure 5

tures.) The results discussed to this point are either published or soon to be so. As a result this summary has been fairly cursive and the manuscripts which describe the data, experimental procedure and details of analysis in considerably more depth are listed in the appendix.

In concluding this section, we discuss some recent results which we feel are of interest and offer some potential for development of isolated structures. These deal with nonisothermal sintering in Fe-Cu. Specifically, we have found that by sintering first at a high sintering temperature and subsequently at a lower one, particles that have coalesced at the higher temperature are fragmented by liquid penetration on exposure to the lower temperature. Optical microscopic examination of specimens sintered only at high temperatures leads one to believe that the solid particles consist of several grains at most. Yet the low temperature treatment indicates that each particle contains many grains, the boundaries across which are apparently low energy since they are difficult to discern by conventional preparation techniques and their apparent  $180^\circ$  dihedral angle where the boundaries originally intersected the particle surface. Thus we currently feel that our difficulty with electron channeling has been a direct result of simultaneous beam focus on several of the "grains" as well as not focusing on truly adjacent grains. We are planning now on focusing on adjacent grains which have become delineated as a result of the low temperature sintering treatment. Finally, we wish to note that the double heat treatment allows the possibility of producing isolated microstructures in fully dense systems by following a high temperature treatment with a low one. Whether such a procedure will serve its purpose depends on the time scale of fragmentation versus subsequent coalescence at low temperature. If the time required for fragmentation of the crystals at low temperatures is less than the time required for subsequent coalescence the process should be feasible and, if vice versa, not feasible. We are presently

conducting such follow-up experiments.

B. Mechanical Fabrication of Fibrous Composites  
from Liquid Phase Sintered Alloys

Results from studies of LPS Fe-Cu alloys indicated that "isolated" solid structures can be developed in LPS alloys which contain minor amounts of the Fe-phase. However, production of such alloys is difficult because of gravity segregation attendant with the formation of isolated structures. However, since iron and copper are mechanically compatible in the sense they can be deformation processed to large strains without fracture of either constituent, deformation processing of isolated LPS alloys in this system offers the intriguing possibility of producing relatively high strength fibrous composites in an economical and straightforward manner. Hence we investigated the processing and properties of such materials in Fe-Cu alloys containing ca. 10-15 vol.% Fe.\*

We have produced starting materials for these composites by two different techniques. The first involves "gentle" arc melting of appropriately blended powder of iron and copper. The temperature can vary from the system peritectic to the liquidus and the resultant microstructure consists of varying amounts of spherical and dendritic iron particles. To produce a consistent blend of these two types of particles by melting at a temperature below the liquidus requires temperature control not readily achieved in arc melting. On the other hand, the vigorous stirring action of the arc prevents segregation from occurring on any macroscopic scale. We also have produced such materials by "directional liquid phase sintering" wherein appropriate powder blends are inserted in alumina tubes and drawn through an induction heating

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\*This composition range does produce isolated structures and so we started originally with these compositions. However, we suspect that higher volume fraction Fe alloys can also be produced in the same manner provided the Fe phase is discontinuous.

coil. Temperature control is much better in this process. However, if the liquid zone is too great in extent, gravity segregation over distances comparable to the extent of this zone can, and does, occur. In summary, the arc melting technique prevents segregation at the expense of particle shape control whereas the situation is vice versa with directional liquid phase sintering.

Alloys so produced can be readily swaged and drawn to fine sizes. Our original studies were conducted as part of Dennis Werth's bachelor project. Yield strengths of alloys so processed are shown as a function of deformation strain in Fig. 6 which also includes similar data for commercially pure copper and data from Bevk et al (8) in Cu-Fe and Cu-Nb alloys originally produced by a somewhat more cumbersome casting technique. As indicated therein, yield strength seems to increase significantly beyond a certain critical deformation strain. In subsequent work we have attempted to define the strengthening mechanisms in composites of this type. In addition to the explicit dependence of volume fraction of iron on composite strength, work hardening (of both species), fiber size and spacing effects on strength of the Fe and Cu respectively, and the interaction between size and work hardening must be considered. Additionally, significant microstructural changes may occur particularly in the "single crystal" iron particles as they are processed. We are currently investigating these effects by controlled thermomechanical processing and recent results are shown in Table I. Tensile strength is a convenient and useful measure of strength in these composites for at least two reasons. First, it is reasonably close to the yield strength since uniform elongation is ca. 2% or less.\* Second, because of the limited ductility,

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\*It is at least a little surprising that these materials which demonstrate so little tensile elongation are capable of extensive mechanical working under multiaxial (including compressive) stress states.

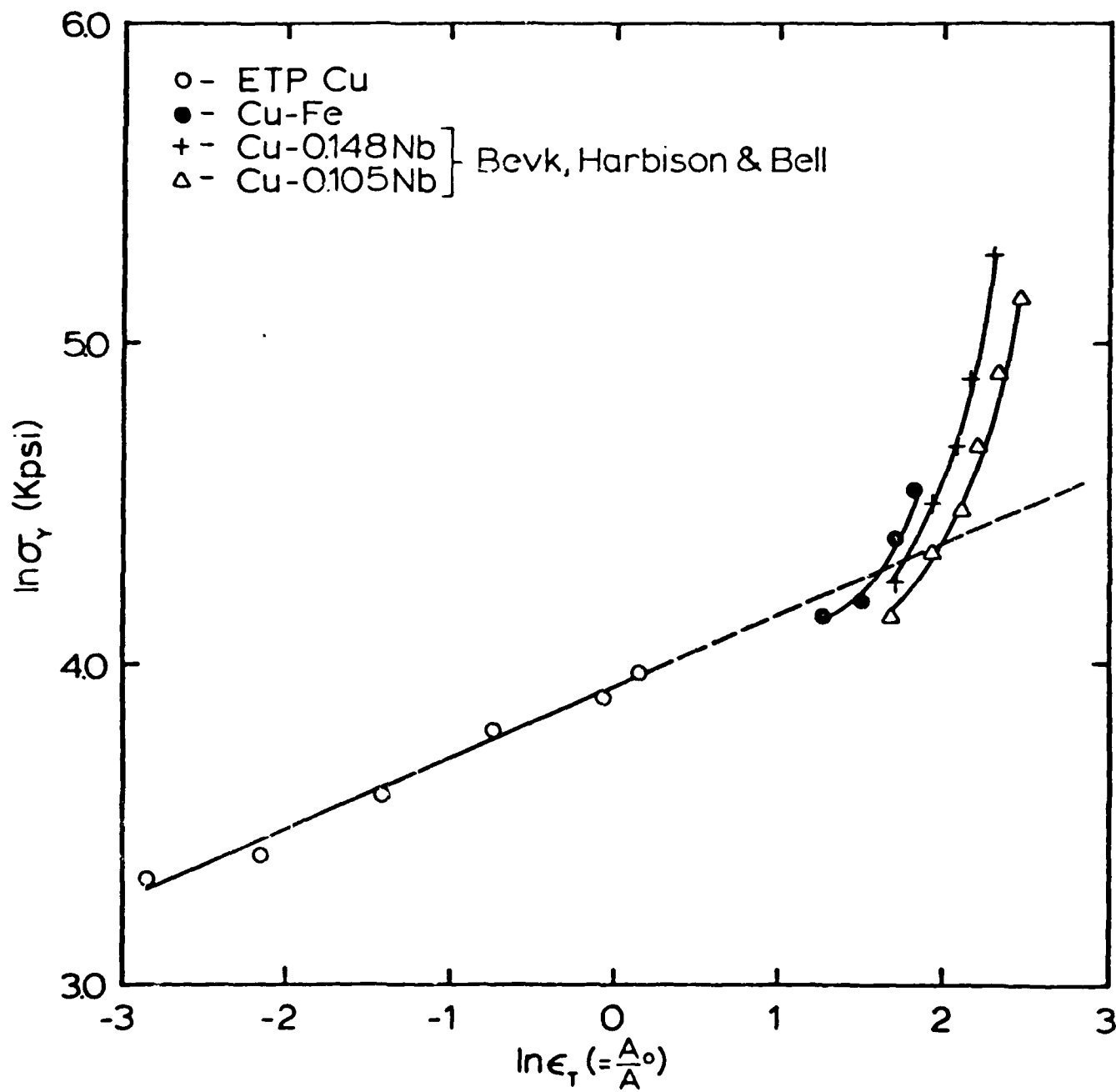


Figure 6

yield strength is not as reproducible from sample to sample as is tensile strength.

We chose to investigate strengthening mechanisms in two ways. First, direct microscopic evidence of structural changes occurring during working is necessary to qualitatively evaluate the importance of, say, work hardening vis-a-vis size effects, and their interaction. This work is continuing. Second, we can phenomenologically relate strength to processing variables. As an example of this latter approach, tensile strength (at a fixed volume fraction of Fe) can be fitted to an equation of the form

$$TS = K_C \epsilon_C^{n_C} + K_F \epsilon_F^{n_F} + K_d d^{-1/2} + \sigma_0, \quad (2)$$

where the first and second terms in Eq. (2) represent (independent) work hardening of both species and the third term represents size effects. With the limited data available, the results of one (not necessarily the optimum) such curve fit of the data to Eq. (2) are shown in Fig. 7 where the experimental and calculated values of tensile strength are compared.\*\* The columns  $\sigma_C$ ,  $\sigma_F$  and  $\sigma_d$  in Table I represent, according to the derived equation, the "independent" contribution of copper ( $K_C \epsilon_C^{n_C}$ ), iron and size effects to the composite strength. According to this model, iron is primarily responsible for the increased composite strength. While the calculated values of tensile strength are within 5%, at worst, of the observed values, we do wish to note that such "curve fitting" is only supplementary to structural studies and that confirmation of, say, the importance of iron in strengthening the composite awaits such information.

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\*In deriving this relationship, it was assumed that a 400°C anneal effectively "recrystallized" the copper but not the iron which only "recrystallizes" after an 800°C anneal. The values of  $\epsilon$  listed in Table I are based on this hypothesis.

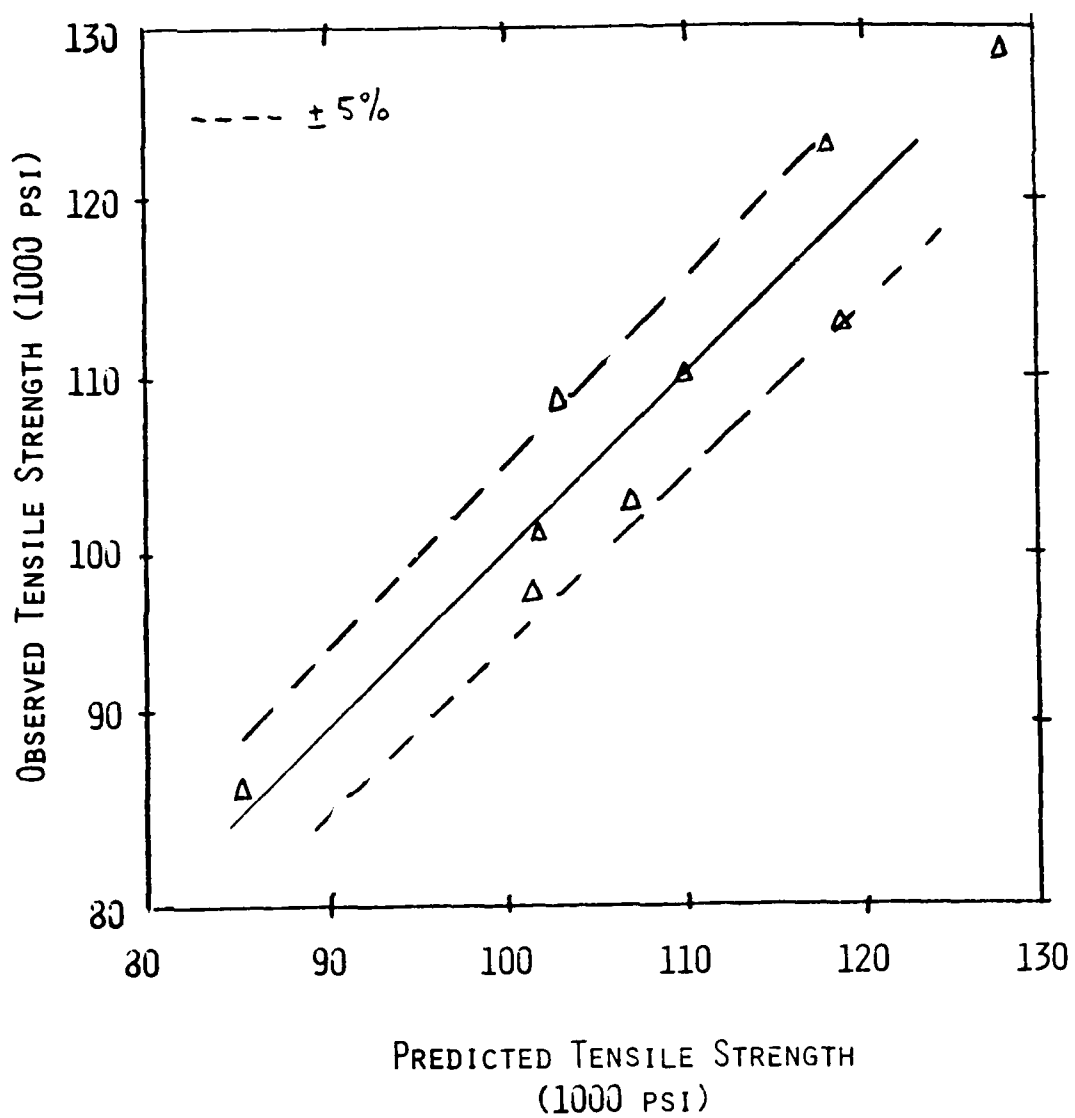


Figure 7

TABLE I

Specimen Code	Processing Schedule <sup>1</sup>	TS(psi)	d(")	$\epsilon_{Cu}$	$\epsilon_{Fe}$	$\frac{\sigma}{Cu}$	$\frac{\sigma}{Fe}$	$\frac{\sigma}{d}$
1	Intermediate Anneal 800°C 2hr @ .0319"	113,000	.010	2.32	2.32	16,300	47,500	42,500
2	Intermediate Anneals 400°C 2hr @ .0284", .0159", .0112", .010"	108,900	.010	0	2.32	0	47,500	42,500
3	Direct Mechanical Fabrication to .0159"	128,800	.0159	5.51	5.51	20,800	60,900	33,700
4	Intermediate Anneal 800°C 2hr @ .0907"	123,000	.0159	3.48	3.48	18,300	53,400	33,700
5	Intermediate Anneal 800°C 2hr @ .0508"	110,100	.0159	2.32	2.32	16,300	47,500	33,700
6	Intermediate Anneal 800°C 2 hr @ .0319"	97,600	.0159	1.39	1.39	14,100	40,900	33,700
7	Intermediate Anneals 400°C 2 hr @ .0284", .0159"	102,800	.0159	0	5.51	0	60,900	33,700
8	Intermediate Anneal 800°C 2hr @ .0907"	100,800	.0284	2.32	2.32	16,300	47,500	25,300
9	Intermediate Anneal 400°C 2hr @ .0284"	86,400	.0284	0	2.32	0	47,500	25,300

1. All material initially processed by "gentle" arc melting followed by swaging to .250". Annealing (at .250") was for 1 hr at 400°C and 1 hr at 800°C. Swaging is then conducted to .140" diameter at which size further deformation processing is by wire drawing.



### Conclusions

In concluding, we reiterate that we feel the main contribution of our efforts has been in identifying and clarifying conditions for development of contiguity and determination of the scale of liquid phase sintered materials. In particular, particle contiguity is essentially only a function of particle volume fraction because of the frequency of (presumably gravity induced) particle collisions during sintering. Microstructural scale is also adversely effected by contiguity. Hence restriction of contiguity and scale can be affected by reducing the latter by 1) production of a "full wetting" system, 2) "trick processing" such as dual isothermal heat treatment, or by 3) reducing substantially the density differences between the solid and liquid phases. In this regard, our current plans include establishing the structural basis of our assumptions by electron channeling of coalesced particles and systematically altering the density differences between the solid and liquid phases during liquid phase sintering.

Our conclusions on the strengthening mechanisms associated with mechanically processed liquid phase sintered alloys are considerably less firm and will be the subject of further work. Although we feel we are on "the right track" in defining such mechanisms, considerably more experimental data and structural analysis are necessary to clearly define the respective mechanisms. Additionally, we plan on defining the role of solid phase contiguity and volume fraction on the possibility of producing such materials by deformation processing.

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## APPENDIX

Graduate Students Supported By This Grant

- M.S. - L. E. Baxa, Degree awarded November, 1980  
A. N. Niemi, Degree awarded November, 1979  
P. Funkenbusch, Degree expected June, 1981
- Ph.D. - A. N. Niemi, Degree expected November, 1981

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